# PATENT SPECIFICATION

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## (54) DETERGENT COMPOSITIONS

(71) We, UNILEVER LIMITED, a company registered under the laws of Great Britain, of Port Sunlight, Wirral, Cheshire, England, do hereby declare the ivention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to detergent compositions, and in particular to detergent compositions including detergency builders.

Eutrophication, which is the process of enrichment of waters with nutrients, can be detrimental since it may cause increased algal growth and hence algal scums which are unaesthetic and clog filters of treatment plants.

Although there is no present adequate proof, it has been speculated that phosphorus-containing and nitrogen-containing detergency builders present in detergent compositions may be a contributing factor in eutrophication. It is, therefore, an object of the present invention to provide detergent compositions with a detergency builder that does not contain nitrogen and phosphorus.

The compositions of the invention necessarily include both a detergency builder and a water-soluble organic detergent active compound. Such detergent active compounds Such detergent active compounds are useful in the present invention are the anionic (soap and nonsoap), nonionic, zwitterionic and ampholytic detergent active compounds. Mixtures can be used. The chemical nature of the detergent active compound is not an essential feature of the present invention. Moreover, such detergent active compounds are well known to those skilled in the detergent art and the patent and printed literature is replete with disclosures of such compounds. Typical of such literature are "Surface Active Agents" by Schwatrz and Perry and "Surface Active Agents and Deter-

gents" by Schwartz, Perry and Berch.

The nitrogen-free and phosphorus-free detergency builders for the detergent compositions of the invention are the normal, that is

the fully neutralised, salts of oxydisuccinic acid (which is also known as 2,2'-oxodisuccinic acid), having the formula:

The preferred salts are the alkali metal, ammonium and water-soluble alkylammonium, e.g. alkanolammonium, salts of oxydisuccinic acid. The salts can also be mixed salts, e.g. monosodium tripotassium oxydisuccinate.

The weight ratio of oxydisuccinate detergency builder to the detergent active compound is preferably from 1:3 to 10:1 and even more preferably from 2:1 to 5:1.

The oxydisuccinate detergency builders can be used either as the sole detergency builders or, where desired, can be used in conjunction with other well-known detergency builders, examples of which include tetrasodium and tetrapotassium pyrophosphate, pentasodium and pentapotassium tripolyphosphate, and trisodium and tripotassium nitrilotriacetate. Other materials which may be present in the detergent compositions of the invention in minor amounts are those conventionally present therein. Typical examples thereof include soil suspending agents, hydrotropes, corrosion inhibitors, dyes, perfumes, fillers, optical brighteners, enzymes, suds boosters, suds depressants, germicides, anti-tarnishing agents and cationic detergents. The balance of the detergent compositions is water.

The detergent compositions of the present invention may be in any of the usual physical forms for such compositions, such as powders, beads, flakes, bars, tablets, liquids and pastes. The compositions are prepared and utilized in

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manner. The wash solutions the conventi thereof desirably have a pH from 7 to 12, preferably from 9 to 11. At pH values below about 8.6 some of the salt of the oxydisuccinic 5 acid will be present in the acid salt form and some in the normal salt form.

The oxydisuccinate detergency builders can be prepared in a conventional manner from oxydisuccinic acid. Thus, for example, tetra-10 sodium oxydisuccinate can be prepared as follows: An aqueous solution containing 5 grams of oxydisuccinic acid (or 2,2'-oxodisuccinic acid) in 35 ml. of distilled water is titrated to a pH of 7.5 with 42 ml. of a 2 N 15 sodium hydroxide solution. The aqueous solvent is removed under reduced pressure and the residue is tetrasodium oxydisuccinate (or tetrasodium 2,2'-oxodisuccinate). The mixed salts of oxydisuccinic acid can be pre-20 pared by neutralizing oxydisuccinic acid with the requisite proportional amounts of basic compounds containing the desired cations.

Examples of the detergent compositions of the invention are set forth below as illustrative but not limitive of such compositions. Percentages are by weight except where other-

wise indicated.

Examples 1-4

The detergent formulations set forth in 30 Table I below (and in Tables II-IX hereinafter) were prepared by blending together the recited components and were then tested for detergency or cleansing ability in the Terg-O-Tometer Test wherein the washing conditions were as follows (unless otherwise indic-

ated): 65% n\*—35% cotton VCD (vacuum cleaner sent) cloth; 120°F.; 180 ppm (2/1 Ca<sup>++</sup>/Mg<sup>++</sup>); 0.15% concentration of the total formulation in the washing solution; pH 9.5. (The pH of the washing solutions given herein was adjusted, where necessary, by the addition of sodium hydroxide thereto).

The average detergency units (DU) of the formulations is the final reflectance of the washed cloth minus the initial reflectance of the soiled cloth (the average of two runs), the reflectance being measured with a Gardner Automatic Color Difference Meter, Model AC--3.

The following abbreviations have been used in Table I (and/or in Tables II—IX): LAS is an anionic surfactant which is sodium linear secondary alkyl (C<sub>10</sub>—C<sub>14</sub>) benzene sulfonate; Neodel 45-11 is a nonionic surfactant which is an adduct of a modified Oxo type C<sub>14</sub>—C<sub>15</sub> alcohol with an average of 11 moles of ethylene oxide;  $C_{14-16}$  HAMT is an ampholytic surfactant which is sodium hydroxyalkyl (C<sub>14</sub>—C<sub>16</sub>) methyltaurate; sulfobetaine is a zwitterionic surfactant which is cocodimethylsulfopropyl betaine; NaODS is tetrasodium oxydisuccinate; STPP is pentasodium tripolyphosphate; TKPP is tetrapotassium pyrophosphate; NTA is trisodium nitrilotriacetate; RU silicate is a sodium silicate having a SiO<sub>2</sub>: Na<sub>2</sub>O weight ratio of 2.4:1; and bal is

TABLE I

	Percent by Weight			
Example No.	1	2	3	4
LAS	18		18	18
NaODS	_	50	_	50
STPP	_		50	-
Water	bal	bal	bal	bal
Average Detergency				
Units (DU):	3.2	2.3	22.5	19.5

<sup>\* &#</sup>x27;Dacron' is a Registered Trade Mark.

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Example 4 compares urably with Example sentative water-soluble 3 which contained ame organic anionic detergent compound and the excellent conventional detergency builder STPP.

Examples 5-8 Examples 5-8, Table II, contained a repre-

c nonionic detergent active compound, lol 45—11. These compositions were prepared and tested in the same manner as set forth above for Examples 1—4 with the exception that the concentration of the total formulation in the washing solution was raised to 0:20% and the pH was 9.3.

TABLE II

Example No:		Percent by Weight			
	5	6	7	8-	
Neodol 45—11	10		10	10	
NaODS	-	50		50	
STPP		_	50		
Water	bal	baľ.	bal:	bal	
Average Detergency Units (DU)	16.6	14.1	26.5	26.1	

15 Examples 9—12 Examples 9-12 presented in Table III below contained a representative water-soluble organic ampholytic detergent active compound identified above. The compositions were pre-

pared and tested in the same manner as set forth for Examples 1-4 except the concentration of the total formulation in the washing solution was 0.20% and the pH was 9.6.

TABLE III

	Percent by Weight				
Example No.	9	10	II	12	
C <sub>14-16</sub> HAMT	18		18	18:	
NaODS		50		<b>50</b> °	
XSTPP			50		
Water	bal	bal	bal:	bal	
Average Detergency Units (DU)	15.1	14.9	26.9	25.9	

25 Examples 13—16 Examples 13—16 given in Table IV below contained a representative water-soluble organic zwitterionic detergent active com-

pound identified above. These compositions were prepared and tested in the same manner as set forth in Examples 5-8.

	Table		rcent by Weight		
Example No.	13	14	15	16	
Sulfobetaine	18		18	18	
NaODS	_	50	_	50	
STPP		440	50		
Water	bal	bal	bal	bal	
Average Detergency Units (DU)	13.5	13.0	26.4	25.6	

Examples 17-20 The formulations of Examples 17—20 in Table V below differ from the preceding Examples 1—16 in that they further contained sodium silicate as a buffer. These compositions were prepared and tested as set forth in the pH was 10.0.

Examples 1-4 except the hardness of the wash water was at three varying concentra-tions, namely 50 ppm, 180 ppm and 360 ppm; the concentration of the total formulation in the washing solution was 0.20%; and

TABLE V

	Percent by Weight			
Example No.	17	18	19	20
Sulfobetaine	18	_	18	
NaODS	_	_	50	50
Neodol 45—11		10	_	10
STPP	50	50		_
RU Silicate · ·	6	6	6	6
Water -	bal ·	bal	bal	bal
Average Detergency Units (DU)	•			
at 50 ppm: at 180 ppm: at 360 ppm:	30.4 27.8 22.4	28.2 - 25.2 17.2	28.3 24.5 19.0	26.7 - 21.8 15.2

Examples 21-23 15

These three examples illustrate the effectiveness of tetrasodium oxydisuccinate as a detergency builder when used in combination with either an inorganic detergency builder (STPP)

or an organic detergency builder (NTA). These compositions were prepared and tested in the same manner as set forth for Examples 17-20 except they were tested at 180 ppm hardness only.

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#### TABLE VI

	Percent by Weight			
Example No.	21	22	23	
Neodol 45—11	10	10	10	
NaODS	-	25	25	
NTA		-	25	
STPP	50	25	_	
Water	bal	bal '	bal	
Average Detergency Units (DU)	25.3	25.2	26.3	

Examples 24-27

These four examples demonstrate the effectiveness of the detergent compositions of the invention in the cleansing of soil cloths other than the 65% Dacron\*—35% cotton VCD soil cloth used in the preceding Examples 1—23. Thus, in Examples 24—27 two differ-

ent further soil test cloths were used, namely, cotton VCD (vacuum cleaner dust) cloth and FDS (Foster D. Snell) soil test cloth. The compositions were prepared and tested as set forth in Examples 21—23 except for the change in the soil test cloths.

TABLE VII

	Percent by Weight				
Example No.	24	25	26	27	
Neodol 45—11	10	-	10	-	
NaODS	-	_	50	50	
Sulfobetaine		18	<del></del>	18	
STPP	50	50			
Water	bal	bal	bal	bal	
Average Detergency Units (DU)					
with cotton VCD	9.9	11.7	9.4	11.0	
with FDS	12.9	16.6	12.8	20.4	

Results similar to those presented hereinabove are achieved when other oxydisuccinate salts are used.

<sup>\*</sup> Registered Trade Mark

AIM IS:-WHAT W

1. A detergent composition containing a water-soluble organic detergent active compound and a detergency builder which is a 5 normal salt of oxydisuccinic acid.

2. A detergent composition as claimed in Claim 1 in which the weight ratio of the salt of oxydisuccinic acid to the detergent active compound is from 1:3 to 10:1.

3. A detergent composition as claimed in Claim 2 in which the weight ratio is from 2:1 to 5:1.

nposition as claimed in 4. A deterger any of claims 1 to 3 in which the salt is the alkali metal, ammonium or alkylammonium

5. A detergent composition according to claim 4 wherein the detergency builder is tetrasodium oxydisuccinate.

6. A detergent composition as described and with particular reference to Examples 4, 8, 12, 16, 19, 20, 22, 23, 26 and 27.

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